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Patent

Case No.: 58354US002

## IN THE UNITED STATES PATENT AND TRADEMARK OFFICE

First Named Inventor:

ROSENFLANZ, ANATOLY Z.

Application No.:

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Group Art Unit:

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Elizabeth Bolden

Title:

METHODS OF MAKING CERAMICS COMPRISING AL<sub>2</sub>O<sub>3</sub>, REO, ZRO<sub>2</sub>

AND/OR HFO2 AND NB2O5 AND/OR TA2O5

## PRELIMINARY AMENDMENT

Mail Stop Non-Fee Amendment Commissioner for Patents P.O. Box 1450 Alexandria, VA 22313-1450

To Fax No.: 703-872-9306

I hereby certify that this correspondence is being facsimile transmitted to the U.S. Patent

CERTIFICATE OF TRANSMISSION

and Trademark Office on:

May 28, 2004

Signed by: Lisa Hengen

Dear Sir:

Please enter the following preliminary amendment in the above-referenced application.

## Amendments to the Specification

Please amend the specification as follows:

On pages 1-41, please delete the page numbering located on the left side of the page.

On page 42, please delete the page numbering located on the right side of the page.

On page 1, please replace the paragraph that starts on line 30 with the word "The" and ends on page 2, line 30, with the word "article" with the following amended paragraph:

In some embodiments, the present invention provides a method for making a glassceramic, the method comprising heat-treating glass to convert at least a portion of the glass to crystalline ceramic and provide glass-ceramic, the glass comprising at least 35 (in some embodiments, at least 40, 45, 50, 55, 60, 65, 70, or even at least 75; in some embodiments, in a range from 35 to 75, 40 to 75, 45 to 75, 50 to 75, 55 to 75, or even from 60 to 75) percent by weight Al<sub>2</sub>O<sub>3</sub>, based on the total weight of the glass, REO (e.g., Gd<sub>2</sub>O<sub>3</sub>, La<sub>2</sub>O<sub>3</sub>, and/or Nd<sub>2</sub>O<sub>3</sub>; in some embodiments, at least 0.5, 1, 2, 3, 4, 5, or even at least 10; in some embodiments, in a range from 0.5 to 70, 1 to 70, 5 to 70, 10 to 70, 10 to 50, 0.5 to 50, 1 to 50, 5 to 50, 10 to 50, 0.5 to 40, 1 to 40, 5 to 40, 10 to 40, 0.5 to 30, 1 to 30, 5 to 30, 10 to 30, 0.5 to 25, 1 to 25, 5 to 25, or even from 10 to 25 percent by weight REO, based on the total weight of the glass), and ZrO2 (in some embodiments, ZrO2 and/or (including collectively) HfO2) (in some embodiments, at least 5, 10, 15, or even at least 20; in some embodiments, in a range from 5 to 30, 5 to 25, 10 to 25, 10 to 30, 15 to 30, 20 to 30, 15 to 25, or even from 15 to 20 percent by weight ZrO<sub>2</sub> (in some embodiments, ZrO<sub>2</sub> and/or (including collectively) HfO<sub>2</sub>), based on the total weight of the glass), and at least one of Nb<sub>2</sub>O<sub>5</sub> or Ta<sub>2</sub>O<sub>5</sub> (in some embodiments, at least 1, 2, 3, 4, 5, 10, 15, 20, or even at least 25; in some embodiments, in a range from 1 to 20, 5 to 20, 10 to 20, or even from 5 to 15 percent by weight at least one of Nb<sub>2</sub>O<sub>5</sub> or Ta<sub>2</sub>O<sub>5</sub>, based on the total weight of the glass), wherein the glass contains not more than 10 (in some embodiments, not more than 9, 8, 7, 6, 5, 4, 3, 2, 1, 0.5, 0.1, or even zero) percent by weight collectively As<sub>2</sub>O<sub>3</sub>, B<sub>2</sub>O<sub>3</sub>, GeO<sub>2</sub>, P<sub>2</sub>O<sub>5</sub>, SiO<sub>2</sub>, TeO2, and V2O5, based on the total weight of the glass, and wherein the at least one of Nb2O5 or Ta<sub>2</sub>O<sub>5</sub> is present in an amount sufficient to increase the rate of at least one of crystalline ZrO<sub>2</sub> or crystalline HfO<sub>2</sub> formation from the glass (in some embodiments, by at least a factor of 1.5, 2, 2.5, or even at least 3 as compared to a comparative glass-ceramic made by heat-treating, in the

same manner, the same glass free of Nb<sub>2</sub>O<sub>5</sub> and Ta<sub>2</sub>O<sub>5</sub> (i.e., the comparative glass is made and heat-treated the same manner as the glass comprising the Al<sub>2</sub>O<sub>3</sub>, REO, at least one of ZrO<sub>2</sub> or HfO<sub>2</sub>, and Nb<sub>2</sub>O<sub>5</sub> and/or Ta<sub>2</sub>O<sub>5</sub> except no Nb<sub>2</sub>O<sub>5</sub> or Ta<sub>2</sub>O<sub>5</sub> was used to make the glass (i.e., such glass contains zero percent by weight Nb<sub>2</sub>O<sub>5</sub> or Ta<sub>2</sub>O<sub>5</sub>, based on the total weight of the glass)). The increased rate of crystalline ZrO<sub>2</sub> and/or crystalline HfO<sub>2</sub> formation from the glass is determined as described below in Example 1. In some embodiments, the method further comprises crushing the glass-ceramic to provide abrasive particles. In some embodiments, the method further comprises grading the abrasive particles to provide a plurality of particles having a specified nominal grade. In some embodiments, the method further comprises incorporating the abrasive particles into an abrasive article.

On page 4, please replace the paragraph that starts on line 19 with the word "Further" and ends on line 27 with the word "oxides)" with the following amended paragraph:

Further, it is understood herein that unless it is stated that a metal oxide (e.g., Al<sub>2</sub>O<sub>3</sub>, complex Al<sub>2</sub>O<sub>3</sub>·metal oxide, etc.) is crystalline, for example, in a glass-ceramic, it may be crystalline, or portions glass and portions crystalline. For example, example if a glass-ceramic comprises Al<sub>2</sub>O<sub>3</sub> and ZrO<sub>2</sub>, the Al<sub>2</sub>O<sub>3</sub> and ZrO<sub>2</sub> may each be in a glass state, crystalline state, or portions in a glass state and portions in a crystalline state, or even as a reaction product with another metal oxide(s) (e.g., unless it is stated that, for example, Al<sub>2</sub>O<sub>3</sub> is present as crystalline Al<sub>2</sub>O<sub>3</sub> or a specific crystalline phase of Al<sub>2</sub>O<sub>3</sub> (e.g., alpha Al<sub>2</sub>O<sub>3</sub>), it may be present as crystalline Al<sub>2</sub>O<sub>3</sub> and/or as part of one or more crystalline complex Al<sub>2</sub>O<sub>3</sub>·metal oxides).

On page 6, please the paragraph that starts on line 5 with the word "In" and ends on page 7, line 2 with the word "article" with the following amended paragraph:

In some embodiments, the present invention provides a method for making abrasive particles, the method comprising heat-treating glass particles to convert at least a portion of the glass to crystalline ceramic and provide glass-ceramic and the abrasive particles, the glass comprising at least 35 (in some embodiments, at least 40, 45, 50, 55, 60, 65, 70, or even at least 75; in some embodiments, in a range from 35 to 75, 40 to 75, 45 to 75, 50 to 75, 55 to 75, or even from 60 to 75) percent by weight Al<sub>2</sub>O<sub>3</sub>, based on the total weight of the glass, REO (e.g., Gd<sub>2</sub>O<sub>3</sub>, La<sub>2</sub>O<sub>3</sub>, and/or Nd<sub>2</sub>O<sub>3</sub>; in some embodiments, at least 0.5, 1, 2, 3, 4, 5, or even at least 10; in some embodiments, in a range from 0.5 to 70, 1 to 70, 5 to 70, 10 to 70, 10 to 50, 0.5 to 50, 1 to 50, 5 to 50, 10 to 50, 0.5 to 40, 1 to 40, 5 to 40, 10 to 40, 0.5 to 30, 1 to 30, 5 to 30, 10 to 30, 0.5 to 25, 1 to 25, 5 to 25, or even from 10 to 25 percent by weight REO, based on the total weight of the glass), ZrO<sub>2</sub> (in some embodiments, ZrO<sub>2</sub> and/or (including collectively) HfO<sub>2</sub>) (in some embodiments, at least 5, 10, 15, or even at least 20; in some embodiments, in a range from 5 to 30, 5 to 25, 10 to 25, 10 to 30, 15 to 30, 20 to 30, 15 to 25, or even from 15 to 20 percent by weight ZrO<sub>2</sub> (in some embodiments, ZrO<sub>2</sub> and/or (including collectively) HfO<sub>2</sub>), based on the total weight of the glass), and at least one of Nb<sub>2</sub>O<sub>5</sub> or Ta<sub>2</sub>O<sub>5</sub> (in some embodiments, at least 1, 2, 3, 4, 5, 10, 15, 20, or even at least 25; in some embodiments, in a range from 1 to 20, 5 to 20, 10 to 20, or even from 5 to 15 percent by weight at least one of Nb<sub>2</sub>O<sub>5</sub> or Ta<sub>2</sub>O<sub>5</sub>, based on the total weight of the glass), wherein the glass contains not more than 10 (in some embodiments, not more than 9, 8, 7, 6, 5, 4, 3, 2, 1, 0.5, 0.1, or even zero) percent by weight collectively As<sub>2</sub>O<sub>3</sub>, B<sub>2</sub>O<sub>3</sub>, GeO<sub>2</sub>, P<sub>2</sub>O<sub>5</sub>, SiO<sub>2</sub>, TeO<sub>2</sub>, and V<sub>2</sub>O<sub>5</sub>, based on the total weight of the glass, and wherein the at least one of Nb<sub>2</sub>O<sub>5</sub> or Ta<sub>2</sub>O<sub>5</sub> is present in an amount sufficient to increase the rate of at least one of crystalline ZrO<sub>2</sub> or crystalline HfO<sub>2</sub> formation from the glass (in some embodiments, by at least a factor of 1.5, 2, 2.5, or even at least 3) as compared to a comparative glass-ceramic made by heat-treating, in the same manner, the same glass free of Nb<sub>2</sub>O<sub>5</sub> and Ta<sub>2</sub>O<sub>5</sub>. In some embodiments, the method further comprises grading the abrasive particles to provide a plurality of particles having a specified nominal grade. In some embodiments, the method further comprises incorporating the abrasive particles into an abrasive article.

On page 7, please the paragraph that starts on line 3 with the word "In" and ends on page line 31 with the word "article" with the following amended paragraph:

In some embodiments, the present invention provides a method for making abrasive particles, the method comprising heat-treating particles comprising glass to convert at least a portion of the glass to crystalline ceramic and provide glass-ceramic and the abrasive particles, the glass comprising at least 35 (in some embodiments, at least 40, 45, 50, 55, 60, 65, 70, or even at least 75; in some embodiments, in a range from 35 to 75, 40 to 75, 45 to 75, 50 to 75, 55 to 75, or from even 60 to 75) percent by weight Al<sub>2</sub>O<sub>3</sub>, based on the total weight of the glass, REO (e.g., Gd<sub>2</sub>O<sub>3</sub>, La<sub>2</sub>O<sub>3</sub>, and/or Nd<sub>2</sub>O<sub>3</sub>; in some embodiments, at least 0.5, 1, 2, 3, 4, 5, or even at least 10; in some embodiments, in a range from 0.5 to 70, 1 to 70, 5 to 70, 10 to 70, 10 to 50, 0.5 to 50, 1 to 50, 5 to 50, 10 to 50, 0.5 to 40, 1 to 40, 5 to 40, 10 to 40, 0.5 to 30, 1 to 30, 5 to 30, 10 to 30, 0.5 to 25, 1 to 25, 5 to 25, or even from 10 to 25 percent by weight REO, based on the total weight of the glass), ZrO2 (in some embodiments, ZrO2 and/or (including collectively) HfO2) (in some embodiments, at least 5, 10, 15, or even at least 20; in some embodiments, in a range from 5 to 30, 5 to 25, 10 to 25, 10 to 30, 15 to 30, 20 to 30, 15 to 25, or even from 15 to 20 percent by weight ZrO<sub>2</sub> (in some embodiments, ZrO<sub>2</sub> and/or (including collectively) HfO<sub>2</sub>), based on the total weight of the glass), and at least one of Nb<sub>2</sub>O<sub>5</sub> or Ta<sub>2</sub>O<sub>5</sub> (in some embodiments, at least 1, 2, 3, 4, 5, 10, 15, 20, or even at least 25; in some embodiments, in a range from 1 to 20, 5 to 20, 10 to 20, or even from 5 to 15 percent by weight at least one of Nb<sub>2</sub>O<sub>5</sub> or Ta<sub>2</sub>O<sub>5</sub>, based on the total weight of the glass), wherein the glass contains not more than 10 (in some embodiments, not more than 9, 8, 7, 6, 5, 4, 3, 2, 1, 0.5, 0.1, or even zero) percent by weight collectively As<sub>2</sub>O<sub>3</sub>, B<sub>2</sub>O<sub>3</sub>, GeO<sub>2</sub>, P<sub>2</sub>O<sub>5</sub>, SiO<sub>2</sub>, TeO<sub>2</sub>, and V<sub>2</sub>O<sub>5</sub>, based on the total weight of the glass, and wherein the at least one of Nb<sub>2</sub>O<sub>5</sub> or Ta<sub>2</sub>O<sub>5</sub> is present in an amount sufficient to increase the rate of at least one of crystalline ZrO2 or HfO2 formation from the glass (in some embodiments, by at least a factor of 1.5, 2, 2.5, or even at least 3) as compared to a comparative glass-ceramic made by heattreating, in the same manner, the same glass free of Nb<sub>2</sub>O<sub>5</sub> and Ta<sub>2</sub>O<sub>5</sub>. In some embodiments, the method further comprises grading the abrasive particles to provide a plurality of particles having a specified nominal grade. In some embodiments, the method further comprises incorporating the abrasive particles into an abrasive article.

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On page 14, please replace the paragraph that starts one line 20 with the word "Some" and ends on line 28 with the word "processing" with the following amended paragraph:

Some embodiments of glasses can also be obtained by other techniques, such as: laser spin melting with free fall cooling, Taylor wire technique, plasmatron technique, hammer and anvil technique, centrifugal quenching, air gun splat cooling, single roller and twin roller quenching, roller-plate quenching, and pendant drop melt extraction (see, e.g., Rapid Solidification of Ceramics, Brockway et al.,[[et. al,]] Metals And Ceramics Information Center, A Department of Defense Information Analysis Center, Columbus, OH, January, 1984). Some embodiments of glasses may also be obtained by other techniques, such as: thermal (including flame or laser or plasma-assisted) pyrolysis of suitable precursors, physical vapor synthesis (PVS) of metal precursors and mechanochemical processing.

On page 15, please replace the paragraph that starts on line 29 with the word "The" and ends on page 16, line 9 with the word "oxide(s)" with the following amended paragraph:

The addition of certain other metal oxides may alter the properties and/or crystalline structure or microstructure of glass-ceramics made according to the present invention, as well as the processing of the raw materials and intermediates in making the ceramic. For example, oxide additions such as CaO, Li<sub>2</sub>O, MgO, and Na<sub>2</sub>O have been observed to alter both the  $T_g$  and  $T_x$  (wherein  $T_x$  is the crystallization temperature) of glass. Although not wishing to be bound by theory, it is believed that such additions influence glass formation. Further, for example, such oxide additions may decrease the melting temperature of the overall system (i.e., drive the system toward lower melting eutectic), and ease glass formation. Compositions based upon complex eutectics in multi-component systems (quaternary, etc.) may have better glass-forming ability. The viscosity of the liquid melt and viscosity of the glass in  $its[its^2]$  working range may also be affected by the addition of metal oxides other than the particular required oxide(s).

On page 21, please replace the paragraph that starts on line 13 with the word "Heat" and ends on line 25 with the word "glasses)" with the following amended paragraph:

On page 22, please replace the paragraph that starts on line 3 with the word "Typically" and ends on line 10 with the word "1979" with the following amended paragraph:

Typically, glass-ceramics are stronger than the glasses from which they are formed. Hence, the strength of the material may be adjusted, for example, by the degree to which the glass is converted to crystalline ceramic phase(s). Alternatively, or in addition, the strength of the material may also be affected, for example, by the number of nucleation sites created, which may in turn be used to affect the number, and in turn the size of the crystals of the crystalline phase(s). For additional details regarding forming glass-ceramics, see, for example example Glass-Ceramics, P.W. McMillan, Academic Press, Inc., 2<sup>nd</sup> edition, 1979.

On page 28, please replace the paragraph that starts on line 20 with the word "In" and ends on page 29, line 4 with the word "JIS10,000" with the following amended paragraph:

In a given particle size distribution, there will be a range of particle sizes, from coarse particles to fine particles. In the abrasive art this range is sometimes referred to as a "coarse", "control" and "fine" fractions. Abrasive particles graded according to industry accepted grading standards specify the particle size distribution for each nominal grade within numerical limits. Such industry accepted grading standards include those known as the American National Standards Institute, Inc. (ANSI) standards, Federation of European Producers of Abrasive Products (FEPA) standards, and Japanese Industrial Standard (JIS) standards. ANSI grade designations (i.e., specified nominal grades) include: ANSI 4, ANSI 6, ANSI 8, ANSI 16, ANSI 24, ANSI 36, ANSI 40, ANSI 50, ANSI 60, ANSI 80, ANSI 100, ANSI 120, ANSI 150, ANSI 180, ANSI 220, ANSI 220, ANSI 240, ANSI 280, ANSI 320, ANSI 360, ANSI 400, and ANSI 600. FEPA grade designations include P8, P12, P16, P24, P36, P40, P50, P60, P80, P100, P120, P150, P180, P220, P320, P400, P500, P600, P800, P1000, and P1200. JIS grade designations include JIS8, JIS12, JIS16, JIS24, JIS36, JIS46, JIS54, JIS60, JIS80, JIS100, JIS150, JIS180, JIS220, JIS240, JIS280, JIS320, JIS360, JIS400, JIS400, JIS600, JIS800, JIS1000, JIS1500, JIS2500, JIS4000, JIS6000, JIS8000, and JIS10,000.

On page 33, please replace the paragraph that starts on line 1 with the word "Grinding" and ends on line 13 with the word "effect" with the following amended paragraph:

Grinding aids encompass a wide variety of different materials and can be inorganic or, organic based. Examples of chemical groups of grinding aids include waxes, organic halide compounds, halide salts and metals and their alloys. The organic halide compounds will typically break down during abrading and release a halogen acid or a gaseous halide compound. Examples of such materials include chlorinated waxes like

tetrachloronaphthalene, tetrachloronaphtalene, pentachloronaphthalene, and polyvinyl chloride. Examples of halide salts include sodium chloride, potassium cryolite, sodium cryolite, ammonium cryolite, potassium tetrafluoroborate, tetrafluoroboate, sodium tetrafluoroborate, silicon fluorides, potassium chloride, and magnesium chloride. Examples of metals include, tin, lead, bismuth, cobalt, antimony, cadmium, and iron titanium. Other miscellaneous grinding aids

include sulfur, organic sulfur compounds, graphite, and metallic sulfides. It is also within the scope of the present invention to use a combination of different grinding aids, and in some instances this may produce a synergistic effect.

On page 33, please replace the paragraph that starts on line 21 with the word "The" and ends on page 34, line 30 with the word "particle" with the following amended paragraph:

The abrasive articles can contain 100% abrasive particles made according to the present invention, or blends of such abrasive particles with other abrasive particles and/or diluent particles. However, at least about 2% by weight, desirably at least about 5% by weight, and more desirably about 30-100% by weight, of the abrasive particles in the abrasive articles should be abrasive particles made according to the present invention. In some instances, the abrasive particles according to the present invention may be blended with another abrasive particles and/or diluent particles at a ratio between 5 to 75% by weight, about 25 to 75% by weight about 40 to 60% by weight, or about 50% to 50% by weight (i.e., in equal amounts by weight). Examples of suitable conventional abrasive particles include fused aluminum oxide (including white fused alumina, heat-treated aluminum oxide and brown aluminum oxide), silicon carbide, boron carbide, titanium carbide, diamond, cubic boron nitride, garnet, fused alumina-zirconia, and sol-gel-derived abrasive particles, and the like. The sol-gel-derived abrasive particles may be seeded or non-seeded. Likewise, the sol-gel-derived abrasive particles may be randomly shaped or have a shape associated with them, such as a rod or a triangle. Examples of sol-gelsol gel abrasive particles include those described in U.S. Pat. Nos. 4,314,827 (Leitheiser et al.), 4,518,397 (Leitheiser et al.), 4,623,364 (Cottringer et al.), 4,744,802 (Schwabel), 4,770,671 (Monroe et al.), 4,881,951 (Wood et al.), 5,011,508 (Wald et al.), 5,090,968 (Pellow), 5,139,978 (Wood), 5,201,916 (Berg et al.), 5,227,104 (Bauer), 5,366,523 (Rowenhorst et al.), 5,429,647 (Larmie), 5,498,269 (Larmie), and 5,551,963 (Larmie). Additional details concerning sintered alumina abrasive particles made by using alumina powders as a raw material source can also be found, for example, in U.S. Pat. Nos. 5,259,147 (Falz), 5,593,467 (Monroe), and 5,665,127 (Moltgen). Additional details concerning fused abrasive particles, can be found, for example, in U.S. Pat. Nos. 1,161,620 (Coulter), 1,192,709 (Tone), 1,247,337 (Saunders et al.), 1,268,533 (Allen), and 2,424,645 (Baumann et al.), et al.) 3,891,408 (Rowse et al.), 3,781,172 (Pett et al.),

3,893,826 (Quinan et al.), 4,126,429 (Watson), 4,457,767 (Poon et al.), 5,023,212 (<u>Dubots et al.</u>), (<u>Dubots et al.</u>), 5,143,522 (Gibson et al.), and 5,336,280 (<u>Dubots et al.</u>), (<u>Dubots et al.</u>), and applications having U.S. Serial Nos. 09/495,978, 09/496,422, 09/496,638, and 09/496,713, each filed on February 2, 2000; 09/618,876, 09/618,879, 09/619,106, 09/619,191, 09/619,192, 09/619,215, 09/619,289, 09/619,563, 09/619,729, 09/619,744, and 09/620,262, each filed on July 19, 2000; 09/704,843, filed November 2, 2000; and 09/772,730, filed January 30, 2001. Additional details concerning ceramic abrasive particles, can be found, for example, in applications having U.S. Serial Nos. 09/922,526, 09/922,527, 09/922,528, and 09/922,530, each filed August 2, 2001, now abandoned, 10/211,597, 10/211,638, 10/211,629, 10/211,598, 10/211,630, 10/211,639, 10/211,034, 10/211,044, 10/211,628, 10/211,491, 10/211,640, and 10/211,684, each filed August 2, 2002; and 10/358,772, 10/358,765, 10/358,910, 10/358,855, and 10/358,708, each filed February 5, 2003. In some instances, blends of abrasive particles may result in an abrasive article that exhibits improved grinding performance in comparison with abrasive articles comprising 100% of either type of abrasive particle.

## <u>REMARKS</u>

Pages 1-42 have been amended to correct editorial errors.

Respectfully submitted,

Data

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